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→ FINAL REPORT
ON TASK 4

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THE THERMAL CONDUCTIVITY OF "M-31"
COATING MATERIAL

NATIONAL AERONAUTICS AND SPACE ADMINISTRATION
GEORGE C. MARSHALL SPACE FLIGHT CENTER
HUNTSVILLE, ALABAMA

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(NASA (Contract NAS8-5196))

(NASA CR-56007)

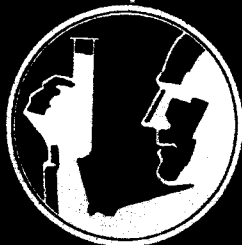
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SOUTHERN RESEARCH INSTITUTE

2000 9th Avenue S.

Birmingham 3, Alabama

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ON TASK 4

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COATING MATERIAL

NATIONAL AERONAUTICS AND SPACE ADMINISTRATION
GEORGE C. MARSHALL SPACE FLIGHT CENTER
HUNTSVILLE, ALABAMA
(Contract NAS8-5196)

Southern Research Institute
Birmingham, Alabama
September 9, 1963
6385-1481-XI

TABLE OF CONTENTS

| | <u>Page</u> |
|---|-------------|
| SCOPE. | 1 |
| SPECIMENS | 1 |
| APPARATUS AND PROCEDURE | 3 |
| DATA AND RESULTS | 3 |
| Heat Flow Normal to the Fibers from 100° F to 800° F | 3 |
| Heat Flow Parallel to the Fibers from 800° F to 1500° F. | 4 |
| Heat Flow Parallel to the Fibers from 100° F to 600° F | 4 |
| Combined Results | 5 |
| APPENDIX. | 22 |
| I. Thermal Conductivity to 1000° F | |
| II. Thermal Conductivity to 5000° F | |

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COATING MATERIAL

SCOPE

The scope of this program was to determine the thermal conductivity in duplicate of an "M-31" coating material (silicone base with potassium titanate fibers) from 100° F to 1500° F (38° C to 816° C).

In an isotropic material, the thermal conductivity will not vary with the direction of the heat flow through the specimen; whereas in anisotropic materials, the thermal conductivity of the specimen varies with the orientation of the material in the apparatus. The "M-31" material was found to be anisotropic depending on the orientation of the heat flow either parallel or perpendicular to the plane of the fibers. Thermal conductivity determinations were made with the "M-31" specimen material oriented in these two primary directions.

The conductivity determinations were made utilizing three different apparatuses to cover the entire temperature range. The results yielded comparable data between the three apparatuses for the same orientation of fiber with heat flow.

SPECIMENS

The specimens were provided by NASA in two configurations. Four specimens were disk-shaped, approximately 7" diameter and $\frac{1}{4}$ " thick with the fibers lengthwise in the lateral plane for evaluation in the guarded hot-plate apparatus; and three specimens were cylindrical, approximately 1" diameter and 1" thick with the fibers lengthwise in a plane perpendicular to the axis of the cylinder for the radial heat flow apparatus.

In the materials, the fibers appeared to have settled, or been forced to settle, toward one surface. The surfaces appearing to have the greater concentration of fibers were more pitted and less uniform than the opposite surfaces. Pits in the specimen surface were ordinarily less than $\frac{1}{16}$ " deep; however, one pit noticed in the surface of one of the cylindrical specimens extended to the inside diameter through a thickness of $\frac{3}{8}$ ".

The specimens evaluated in the guarded hot-plate apparatus were oriented with the heat flowing from the smoother surface to the rougher surface, from lesser to greater concentration of fibers. The cylindrical specimens appeared to have a rather uniform concentration of fibers through the radius of the specimens with some variation axially.

The specimens were rather difficult to machine. The fibers tended to cause the specimen material to flake and separate on the surface, resulting in a surface with a fuzzy texture. No final machining was required for the 7" diameter, guarded hot-plate specimens.

The surfaces of those specimens exposed to relatively high temperatures began degrading rather rapidly at approximately 2000° F (1093° C). The remainder of the specimens did not appear to be affected by their lower temperature exposures.

Two of the 7" diameter specimens were cut in strips; the strips were turned on edge and glued with "Silastic" RTV 731. The surfaces were smoothed and two 3" diameter specimens approximately $\frac{1}{4}$ " thick were cut out. Thermal conductivity evaluations of these specimens were made from 100° F to 600° F (38° C to 316° C), with heat flowing through the thickness of the specimen and parallel to the fibers. This procedure, coupled with the others, provided evaluations from 100° F to 800° F of the material with the heat flow both normal and parallel to the fibers and evaluations from 800° F to 1500° F with the heat flow parallel to the fibers. Figure 1 is a photograph of the two types of specimens evaluated in the modified ASTM C 177-45 guarded hot-plate apparatuses.

The glue between the strips of the fabricated 3" diameter specimens created an interface which was ordinarily not greater than 0.008" thick. An interface thickness of 0.010" was assumed for an error analysis which indicated that the area of the specimen occupied by the interfaces was only 4.07% of the total effective specimen area. Reported values for the thermal conductivity of similar cements are normally in the range of 0.8 to 1.5 Btu/hr/ft²/° F/in., so the maximum error introduced by the difference in conductivity of the glue and specimen material would be 3%.

APPARATUS AND PROCEDURE

The thermal conductivity of the "M-31" material normal to the fibers was evaluated from 100° F to 800° F (38° C to 427° C) in 100° F increments in the 7" diameter guarded hot-plate apparatus. This apparatus is a modification of the standard ASTM C 177-45 design. A complete description is attached as Appendix I. The first set of evaluations were made using Fiberfrax as a filler material throughout the temperature range. The second set of evaluations was made with gum rubber filler at 100° F and Fiberfrax through the remainder of the exposure. All evaluations made in the 7" diameter rig were with the heat flowing normal to the specimen and normal to the fibers.

From 800° F to 1500° F (427° C to 816° C), the thermal conductivity was determined by a direct measurement technique in the radial heat flow apparatus. A description of this apparatus and procedure is attached as Appendix II, "Thermal Conductivity to 5000° F." Two specimens of the design shown in Figure 2 were evaluated in temperature intervals of approximately 200° F. For each of these evaluations, the heat was flowing on the radius of the specimen or parallel with the plane of the lengths of the fibers. It is assumed that the fibers were randomly oriented in the plane.

A third apparatus was utilized to evaluate the "M-31" material from 100° F to 600° F (38° C to 316° C) with the heat flowing in the thickness direction of the specimen, but parallel to the plane of the lengths of the fibers. This apparatus was a 3" diameter guarded hot-plate design similar to the 7" diameter rig with an effective heating radius of 0.95". Specimens were fabricated from the "M-31" material evaluated in the 7" diameter apparatus as described. Fiberfrax and gum rubber were used for filler material in these evaluations.

DATA AND RESULTS

Heat Flow Normal to the Fibers from 100° F to 800° F

Using the 7" guarded hot plate, the thermal conductivity of the first specimen of "M-31" material increased in a direction normal to the fibers from 0.85 Btu/hr/ft²/° F/in. at 100° F to 1.30 Btu/hr/ft²/° F/in. at 730° F, as shown in Figure 3. This increase occurred gradually over

the entire temperature range. Figure 4 shows the conductivity of the second "M-31" specimen with the same orientation evaluated from 100° F to 800° F, and Figure 5 is a comparison of the results of the two evaluations. Tabulated results are given in Tables 1 and 2.

During the evaluation of the first specimen, the thermocouples on the hot surface of the specimens failed at approximately 800° F. Post-exposure inspection revealed that the brass "getters" attached to the thermocouples had been attacked chemically. A sheet of commercial grade aluminum foil was placed between the specimen surface and thermocouples, and the evaluations were continued to a specimen mean temperature of 730° F. A second measurement of the conductivity at 500° F revealed that the aluminum foil had no deleterious effect on the results.

Heat Flow Parallel to the Fibers from 800° F to 1500° F

Using the radial heat flow apparatus, two cylindrical specimens were evaluated with the results shown in Figure 6 and Tables 3 and 4. Parallel to the fibers, the conductivity of the first specimen was constant through the temperature exposure range at 1.7 Btu/hr/ft²/° F/in.; whereas the conductivity of the second specimen, oriented similarly, decreased from 1.86 Btu/hr/ft²/° F/in. at 800° F to 1.43 Btu/hr/ft²/° F/in. at 1500° F. Figure 7 is a comparison of the conductivities of the two specimens.

The specimen material appeared to degrade considerably at temperatures above 2000° F. Figure 8 is a photograph of the two specimens evaluated in the radial heat flow apparatus and shows the serious deterioration of the bottom guards and portions of the specimens. The furnace was purged with helium.

Proper isothermal conditions were maintained by centering the specimen in the furnace and allowing the apparatus to reach a steady-state condition. Figure 9 is a photograph of the isothermal lines on the top of one of the specimens after evaluation in the radial heat flow apparatus. There was some disruption of the isothermals axially, but calculation shows this error to be less than 1%.

Heat Flow Parallel to the Fibers from 100° F to 600° F

Using the 3" hot plate, an evaluation of the thermal conductivity of the specimen material was made from 100° F to 600° F with the heat flowing

through the material parallel to the lengths of the fibers. The specimen was fabricated from strips of the specimen material previously evaluated to 730° F. The results of this evaluation are shown in Figure 10 and Table 5. The conductivity of this specimen remained very constant at 1.73 Btu/hr/ft²/° F/in. through the exposure range. Gum rubber was used for a filler material for the 100° F determination and Fiberfrax was used for the remainder. Changing the filler material had no effect on the results.


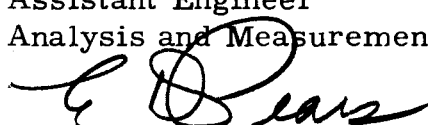
No correction was made to the data obtained for the composite specimen of "M-31" material and "Silastic" glue due to the small proportion of the total area occupied by the glue.

Combined Results


Figure 11 shows the combined results of the thermal conductivity of the material using the three apparatuses and two primary directions of heat flow. The conductivity of the material with heat flowing in a direction parallel to the lengths of the fibers was essentially constant at 1.7 Btu/hr/ft²/° F/in. from 100° F to 1500° F. The conductivity of the material was 0.85 Btu/hr/ft²/° F/in. at 100° F and increased to 1.3 Btu/hr/ft²/° F/in. at 730° F when heat was flowing perpendicular to the plane of the lengths of the fibers.

The results indicated that the "M-31" coating material was anisotropic in composition. The conductivity varied with material orientation and was relatively constant from 100° F to 1500° F in the plane of the lengths of the fibers.

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Figure 1. Photograph of "M-31" Coating Material Specimens Before Evaluation in the Modified ASTM C 177-45 Guarded Hot-Plate Apparatuses.

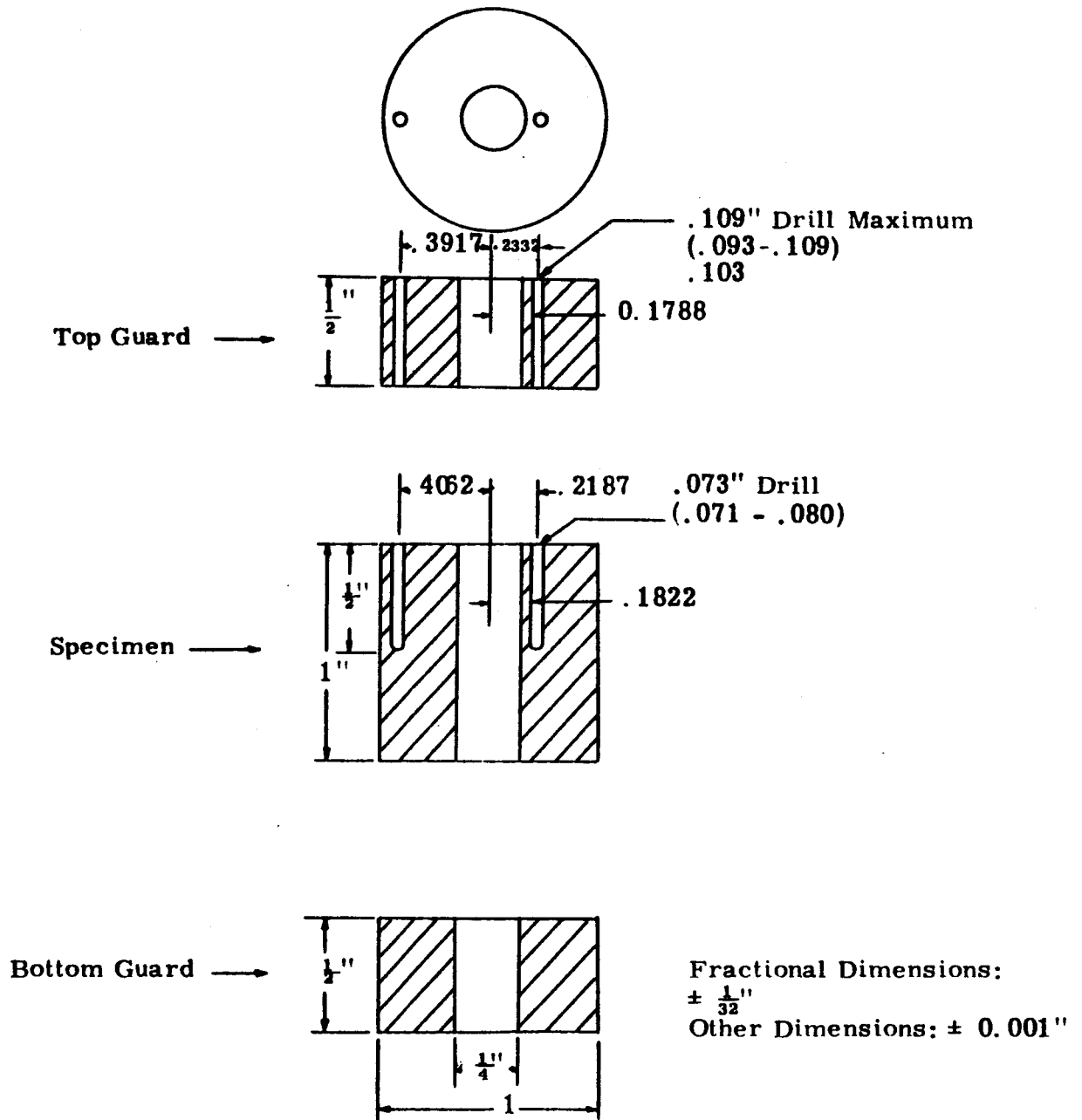


Figure 2. Preferred Configuration of the Cylindrical Specimens for Thermal Conductivity

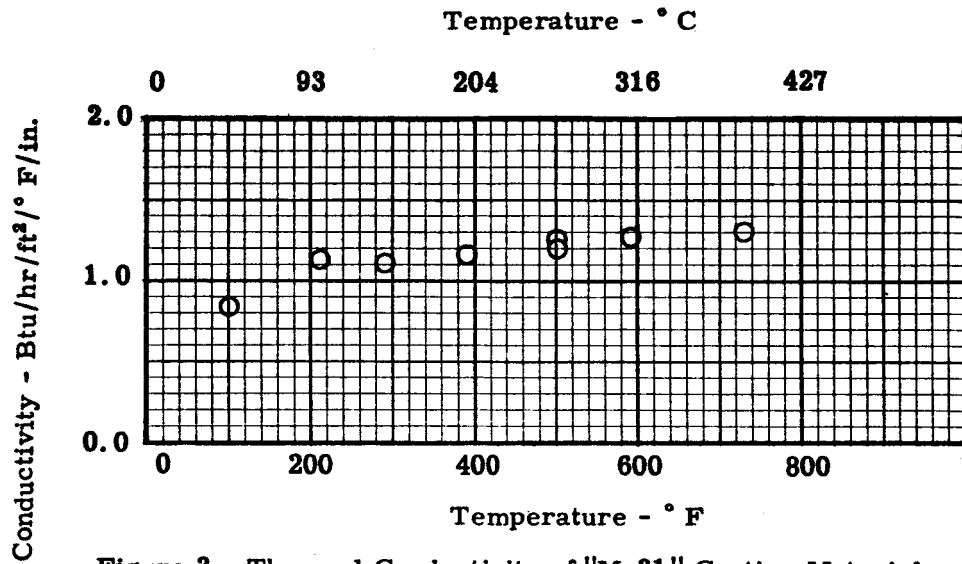


Figure 3. Thermal Conductivity of "M-31" Coating Material During the First Run on the 7" Dia Modified ASTM C 177-45 Apparatus - Specimen No. 1 (Heat Flow Normal to Fibers)

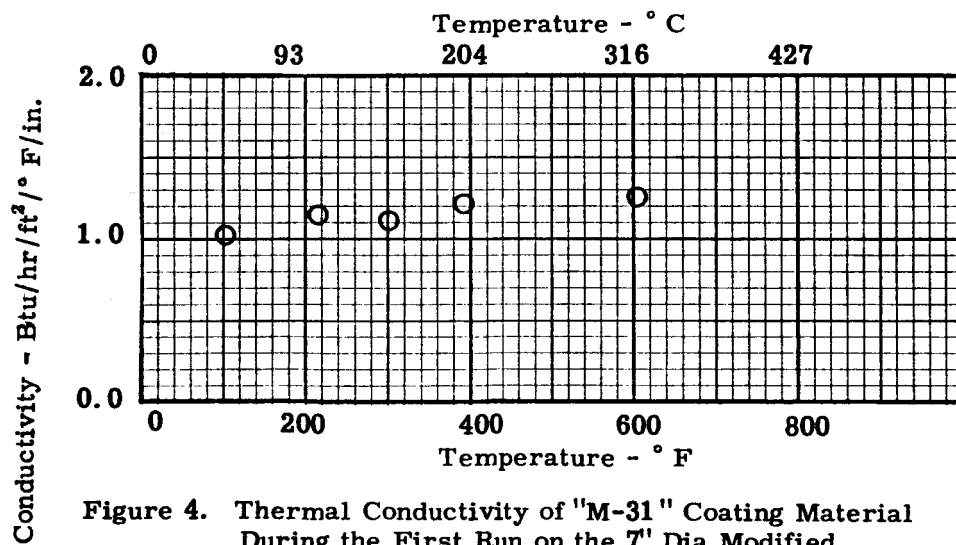


Figure 4. Thermal Conductivity of "M-31" Coating Material During the First Run on the 7" Dia Modified ASTM C 177-45 Apparatus - Specimen No. 2 (Heat Flow Normal to Fibers)

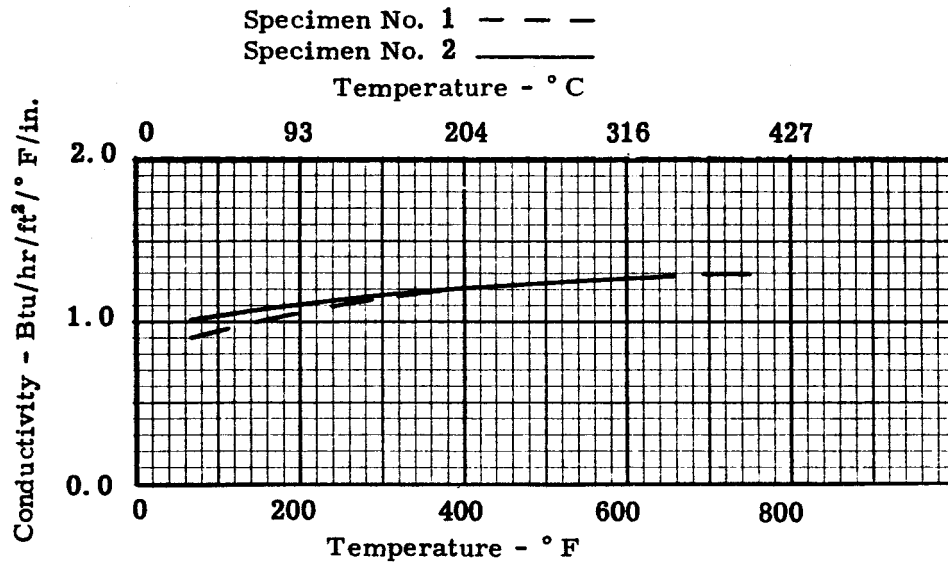


Figure 5. Comparison of Conductivity of Two "M-31" Specimens During the First Run on Each in the 7" Dia Modified ASTM C 177-45 Apparatus. (Heat Flow Normal to the Fibers)

Specimen No. 1 O
Specimen No. 2 Δ

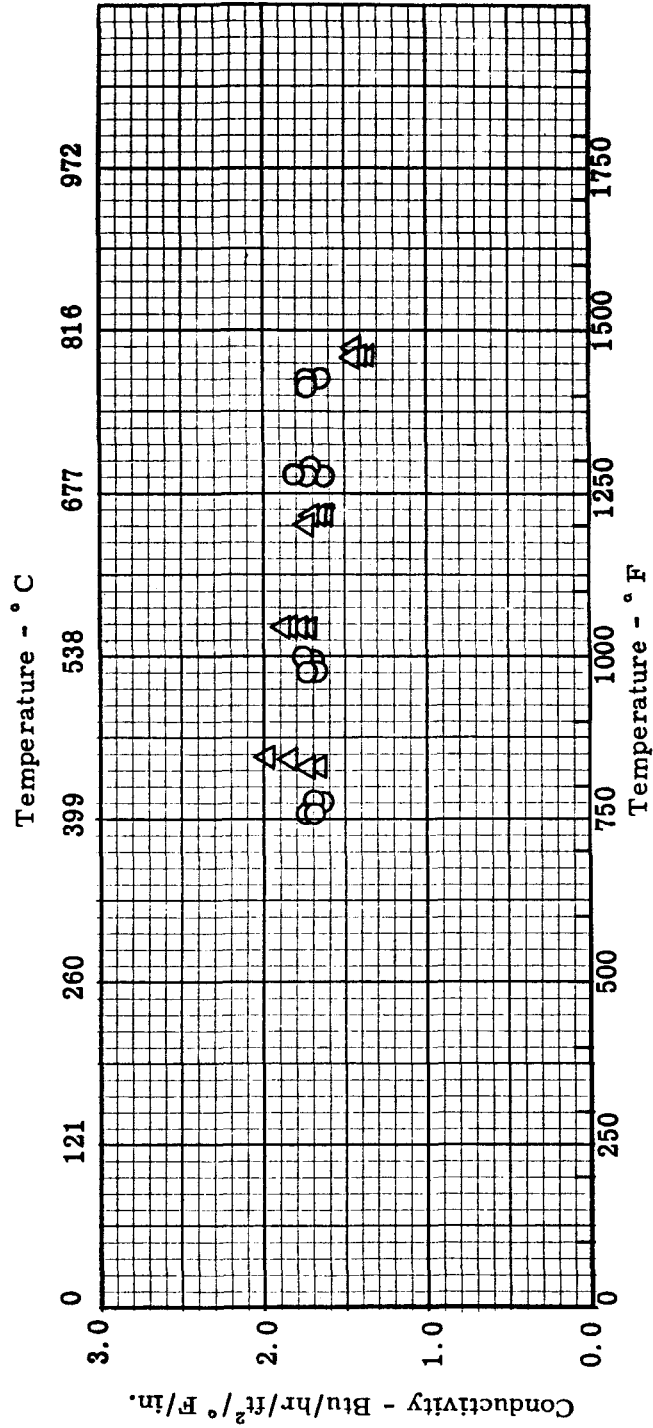


Figure 6. Thermal Conductivity of Two "M-31" Coating Material Specimens During the First Run for Each in Radial Heat Flow Apparatus. (Heat Flow Parallel to the Fibers)

Specimen No. 1 ———
 Specimen No. 2 - - -

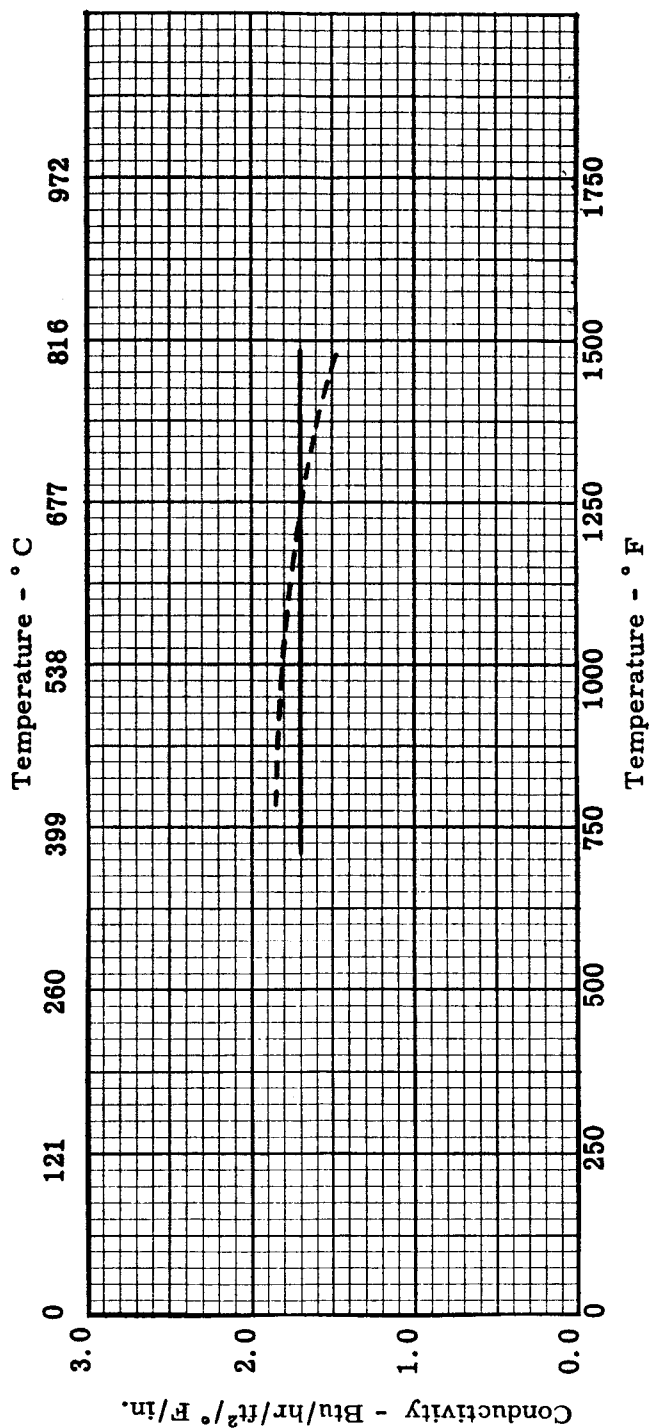


Figure 7. Comparison of the Thermal Conductivities of Two "M-31" Coating Material Specimens During the First Run for Each in the Radial Heat Flow Apparatus. (Heat Flow Parallel to the Fibers)



Figure 8. Photograph of Two "M-31" Coating Material Specimens After Evaluation to Different Maximum Temperature in the Radial Heat Flow Apparatus.

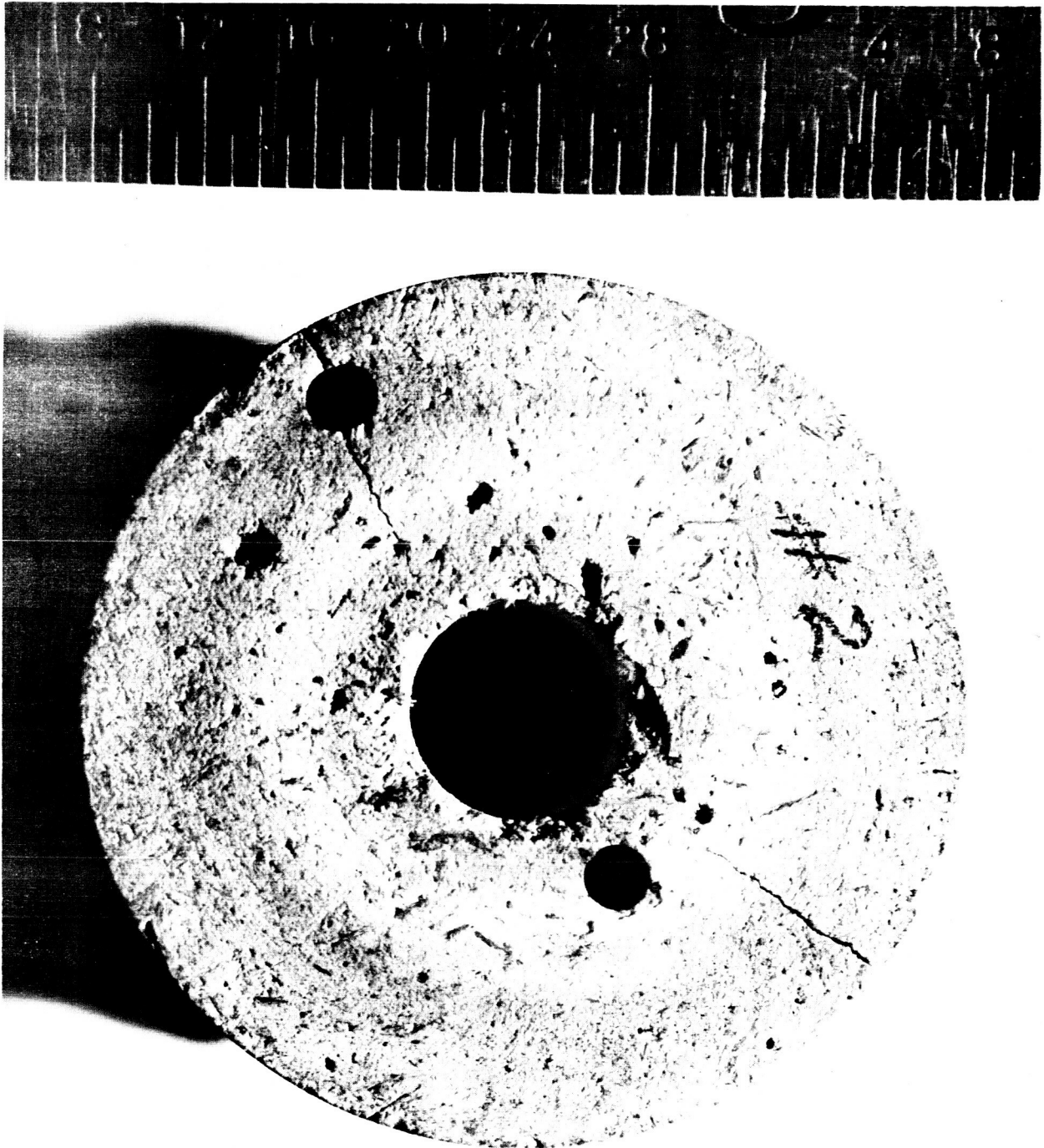


Figure 9. Photograph of Isothermal Lines on the Top Surface of an "M-31" Coating Material Specimen Evaluated in the Radial Heat Flow Apparatus.

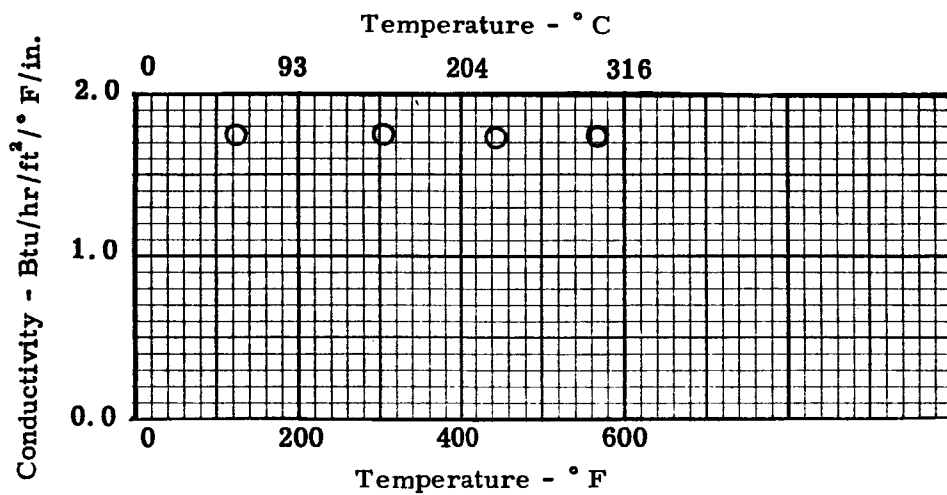


Figure 10. Thermal Conductivity of Fabricated "M-31" Specimen Material During the First Run in the 3" Dia Modified ASTM C 177-45 Apparatus. (Heat Flow Parallel to the Fibers)

Normal to Fibers - 7" Dia Apparatus
 Parallel to Fibers - 3" Dia Apparatus to 600° F
 Parallel to Fibers - Radial Heat Flow Apparatus, 700° F to 1500° F

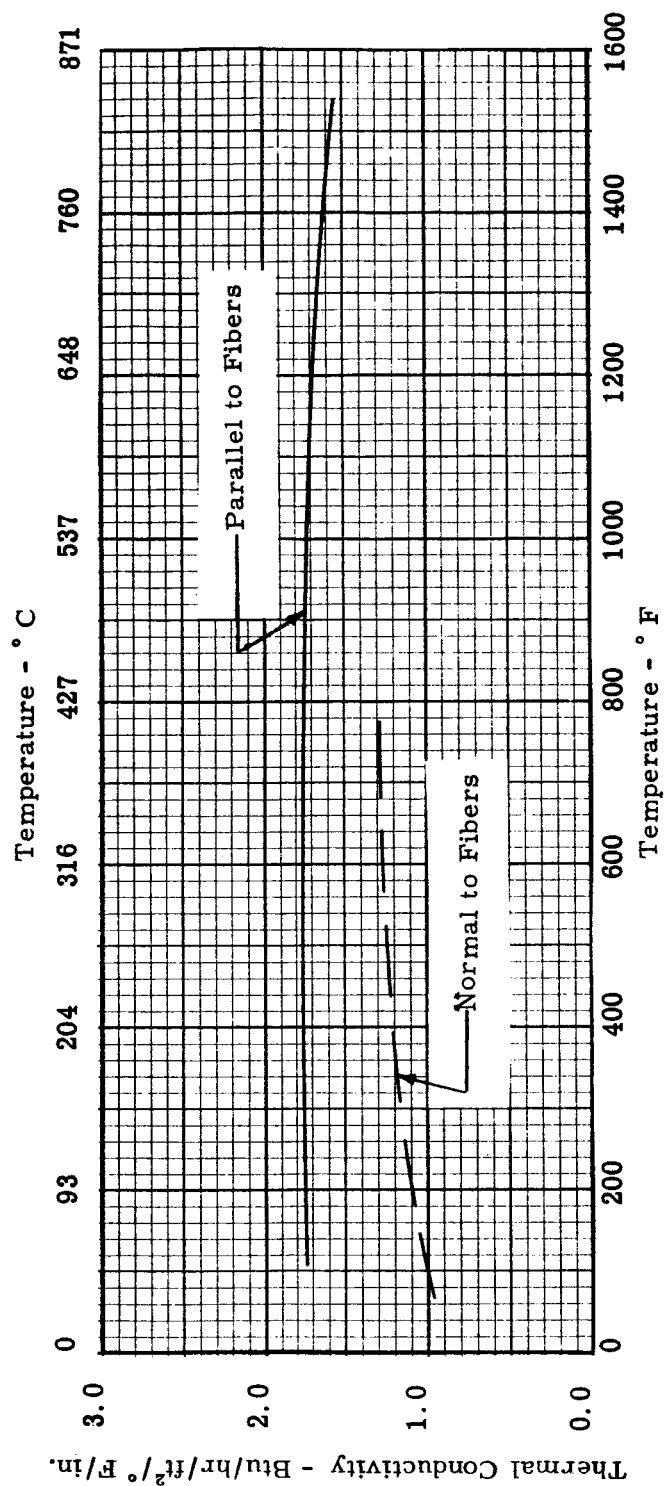


Figure 11. Thermal Conductivity of "M-31" Coating Material Normal and Parallel to the Plane of the Lengths of the Fibers.

Table 1

Thermal Conductivity of "M-31" Coating Material During the First Run on the 7" Diameter Modified ASTM C 177-45 Apparatus¹ (Specimen No. 1)

| Average Specimen Mean Temperature °F | Total Heat Input Watts | Average Specimen ΔT °F | Specimen Thermal Conductivity Btu/hr/ft ² /°F/in. | Remarks |
|--------------------------------------|------------------------|--------------------------------|--|---|
| 98.48 | 2.15 | 12.35 | 0.85 | Fiberfrax filler |
| 212.21 | 20.93 | 90.22 | 1.13 | |
| 293.43 | 33.70 | 147.10 | 1.11 | |
| 388.78 | 51.40 | 212.73 | 1.17 | |
| 502.08 | 74.70 | 298.50 | 1.21 | |
| 503.15 | 52.19 | 204.17 | 1.24 | Brass temperature sensors reacted with specimen material at about 800°F |
| 593.63 | 65.71 | 250.35 | 1.28 | Specimen in foil and insulation between cold plate and cooling section |
| 729.46 | 86.60 | 325.08 | 1.30 | |

Mean specimen thickness = 0.24864 in.

¹ Heat flow normal to the fibers.

Table 2

Thermal Conductivity of "M-31" Coating Material During the
First Run on the 7" Diameter Modified ASTM C 177-45 Apparatus
(Specimen No. 2)
(Heat Flow Normal to the Fibers.)

| Average Specimen Mean Temperature °F | Total Heat Input Watts | Average Specimen ΔT °F | Specimen Thermal Conductivity Btu/hr/ft ² /°F/in. | Remarks |
|--------------------------------------|------------------------|--------------------------------|--|---|
| 102.56 | 2.76 | 13.24 | 1.01 | Gum rubber filled |
| 216.28 | 11.07 | 46.35 | 1.15 | Fiberfrax filler and asbestos between cold plate and cooling section. Specimen in foil. |
| 302.95 | 16.95 | 73.86 | 1.11 | |
| 394.57 | 25.27 | 100.71 | 1.21 | |
| 604.58 | 42.78 | 161.93 | 1.27 | |

Mean specimen thickness = 0.24673 in.

Table 3
Thermal Conductivity of "M-31" Coating Material During the First Run
in the Radial Heat Flow Apparatus (Specimen No. 1)
(Heat Flow Parallel to the Fibers)

| SRI Run Number | Time | Specimen Outer Face Temperature °F | ΔT Across $\frac{3}{8}$ " Specimen Gage Length °F | Total Heat Removed by $\frac{1}{2}$ " Calorimeter Gage Length Btu/hr | Mean Temperature of Specimen °F | Specimen Thermal Conductivity Btu/hr/ft ² /°F/in. |
|----------------|------------------------------|------------------------------------|---|--|---------------------------------|--|
| 1 | On 4:00 Read 8:46 | - | 423 | 24.64 | 776 | 1.65 |
| | | - | 423 | 25.20 | 776 | 1.69 |
| | | - | 423 | 25.48 | 757 | 1.71 |
| | | - | 423 | 25.93 | 756 | 1.74 |
| | Up 9:04 Read 10:19 | - | 560 | 34.20 | 975 | 1.73 |
| | | - | 560 | 33.76 | 976 | 1.71 |
| | | - | 560 | 35.45 | 996 | 1.78 |
| | | - | 560 | 34.63 | 993 | 1.76 |
| | Up 10:40 Read 11:34 | - | 737 | 45.07 | 1276 | 1.74 |
| | | - | 737 | 45.19 | 1277 | 1.74 |
| | | - | 737 | 47.00 | 1276 | 1.81 |
| | | - | 737 | 42.67 | 1275 | 1.64 |
| | Up 11:43 | - | | | | |

Table 3 Continued
Thermal Conductivity of "M-31" Coating Material During the First Run
in the Radial Heat Flow Apparatus (Specimen No. 1)
(Heat Flow Parallel to the Fibers)

| SRI Run Number | Time | Specimen Outer Face Temperature °F | ΔT Across $\frac{3}{16}$ " Specimen Gage Length °F | Total Heat Removed by $\frac{1}{8}$ " Calorimeter Gage Length Btu/hr | Mean Temperature of Specimen °F | Specimen Thermal Conductivity Btu/hr/ft ² /°F/in. |
|----------------|------------|------------------------------------|--|--|---------------------------------|--|
| 1 (cont'd) | Read 12:34 | - | 816 | 47.67 | 1425 | 1.66 |
| | | - | 816 | 49.74 | 1418 | 1.73 |
| | | - | 816 | 49.74 | 1418 | 1.73 |
| | Off 12:47 | - | 816 | 49.73 | 1417 | 1.73 |

Table 4
Thermal Conductivity of "M-31" Coating Material During the First Run
in the Radial Heat Flow Apparatus (Specimen No. 2)
(Heat Flow Parallel to the Fibers)

| SRI Run Number | Time | Specimen Outer Face Temperature °F | ΔT Across 3" Specimen Gage Length °F | Total Heat Removed by 1" Calorimeter Gage Length Btu/hr | Mean Temperature of Specimen °F | Specimen Thermal Conductivity Btu/hr/ft ² /°F/in. |
|----------------|------------|------------------------------------|--|---|---------------------------------|--|
| 2 | On 9:15 | | | | | |
| | Read 10:27 | - | 493 | 29.52 | 823 | 1.70 |
| | | - | 495 | 28.13 | 824 | 1.61 |
| | | - | 468 | 31.52 | 838 | 1.91 |
| | Up 10:44 | - | 469 | 34.03 | 840 | 2.02 |
| | Read 12:32 | - | 610 | 41.36 | 1038 | 1.93 |
| | | - | 612 | 40.88 | 1041 | 1.90 |
| | | - | 625 | 39.23 | 1040 | 1.78 |
| | Up 12:47 | - | 627 | 38.11 | 1041 | 1.73 |
| | Read 1:44 | - | 801 | 47.98 | 1210 | 1.70 |
| | | - | 801 | 46.52 | 1206 | 1.65 |
| | | - | 804 | 50.47 | 1202 | 1.78 |
| | Up 1:58 | - | 802 | 49.15 | 1199 | 1.74 |
| | Read 2:50 | - | 963 | 49.59 | 1463 | 1.46 |
| | | - | 961 | 48.37 | 1463 | 1.43 |
| | | - | 959 | 48.83 | 1463 | 1.45 |
| | | - | 962 | 45.71 | 1457 | 1.35 |
| | Off 3:07 | | | | | |

Table 5

Thermal Conductivity of "M-31" Coating Material During the
First Run on the 3" Diameter Modified ASTM C 177-45 Apparatus
(Heat Flow Parallel with the Fiber.)

| Average Specimen Mean Temperature °F | Total Heat Input Watts | Average Specimen ΔT °F | Specimen Thermal Conductivity Btu/hr/ft ² /°F/in. | Remarks |
|--------------------------------------|------------------------|--------------------------------|--|-------------------|
| 121.79 | 2.30 | 30.38 | 1.73 | Gum rubber filler |
| 304.91 | 6.93 | 91.02 | 1.74 | Fiberfrax filler |
| 442.18 | 11.05 | 145.06 | 1.74 | |
| 564.06 | 14.57 | 192.52 | 1.73 | |

Mean specimen thickness = 0.26420 in.

APPENDIX

Appendix No. I: Thermal Conductivity to 1000° F

Appendix No. II: Thermal Conductivity to 5000° F

THERMAL CONDUCTIVITY TO 1000°F

Thermal conductivity runs are made with a guarded hot plate, which is a slight modification of the standard ASTM C 177-45 design.

The apparatus consists of a central heater plate surrounded by a guard heater, each separately controlled. The guard ring is maintained at the same temperature as the central heater so that all of the heat flow is normal to the specimen surfaces. The temperature difference between the guard and central sections is measured by means of eight differential-thermocouple junctions connected in series. The plate containing the two heaters is sandwiched between layers of sheet insulation, the hot-face thermocouples, the specimen, cold-face thermocouples, sheet insulation, a copper plate, and finally a cold source to dissipate the heat. The cold source consists of a copper coil enclosed in an aluminum box. In addition to the thermocouples in contact with the specimen, thermocouples are located in the central heater and the outer copper plates.

The thermocouples on the hot side and the cold side are sandwiched between sheets of thin asbestos paper. The ends of the thermocouple leads protrude through one sheet of the paper and are soldered to 1" x 1" squares of brass shim stock. This arrangement insures that there is no air film between the specimen and the thermocouples as well as between the specimen and the hot and the cold plate. The use of the paper and thermocouple getters increases the flexibility of the apparatus for use with materials of varying surface finishes; however, for materials with unusual surface finishes, neoprene sheets are inserted on both sides of the specimens. The neoprene deforms to the surface of the specimen thus eliminating the air film. The apparent limitation of this arrangement is the destruction temperature of the neoprene.

Single thermocouples in the center of the heater plate monitor the heater temperature. In addition, five thermocouples are in the cold copper plates. Four of the thermocouples protrude through the plate in contact with asbestos sheets. The fifth thermocouple is soldered to the cold plate to monitor its temperature. These couples, in the heater and the cold plates, are used to monitor the over-all temperature drop through the assembly.

To maintain good contact pressure, a screw loading device holds the entire sandwich assembly pressed firmly together with a total load application up to about 600 pounds.

The assembly is arranged to operate with the specimen placed in the apparatus horizontally as shown in Figure 1. The assembly is insulated around the edges by glass batting, which can be seen on the far sides of the apparatus in Figure 1.

A constant voltage transformer is used in conjunction with the variable control transformers to assure a constant power supply at each setting. The central heater and guard heater are controlled individually by the variable control transformers. The voltage and current to the central heater are monitored by means of a voltmeter and an ammeter, which are switched out of the circuit except when actually being read. The voltage to the guard heater is monitored constantly by a voltmeter.

All of the thermocouple readings are taken on a Leeds and Northrup K-2 potentiometer in conjunction with a galvanometer of 0.43 microvolts per mm deflection sensitivity.

To obtain mean sample temperatures above room temperature, water is circulated through the copper tubing of the cold plates. For mean sample temperatures below room temperature, cold trichloroethylene is pumped through the copper tubing. This coolant is chilled by circulating it through copper coils in a trichloroethylene dry-ice bath. Equilibrium conditions are certified before readings are taken.

Data and Results

Coefficients of thermal conductivity are calculated from the expression:

$$K = \frac{QX}{A\Delta t}$$

where: Q = total heat flow - Btu/hr

X = average thickness of specimens - inches

A = area of central heater section - square feet

Δt = sum of temperature drops across each sample - °F

Theoretically, Q, the heat input, should split, with exactly half of the input flowing through each sample. The temperature drops indicate that this condition rarely exists. Instead, there is a slight unbalance in the heat flow. The above formula then permits a calculation of the arithmetic average for the two panels.

As a check, the thermal conductivity can be calculated for the specimen with a series expression, knowing the over-all temperature drop from the heater to the cold sink and the conductivity of the asbestos and/or the neoprene.

The original calibration curve from early work on the conductivity apparatus is shown in Figure 2. Several data points also are included that were obtained on some reference specimens at the start of one job to check out the use of smaller specimens, a modified periphery insulation, and an improved temperature measuring technique. From this curve, it can be seen that the data had considerable scatter. In spite of this scatter, sufficient information was obtained to establish operation procedure and techniques and to confirm the validity of using smaller specimens.

A more accurate calibration curve was established and is shown in Figure 3. From this data it was determined that the best operating procedure was to measure the face temperature with five thermocouples mounted on small brass "getters" and held against the specimen by a thin sheet of asbestos paper. Also, the copper plates on the hot side were eliminated. These procedures produced data, which practically duplicated the previous calibrations on 14" specimens with 6" vermiculite insulation around the apparatus. With copper plates on the hot side, the conductivity obtained was higher, indicating a radial heat loss out through the copper plates on the heater side of the specimens.

Recent calibration of the apparatus had been with plexiglas as a reference specimen. The results of the calibration runs are shown in Figure 4, including reference data by other investigators. The excellent agreement further established the reliability of the equipment.

The present procedure, which includes thermocouples soldered to brass "getters" on both sides of the specimen, was also calibrated with the plexiglas specimen. The results, also shown in Figure 4, established the latter procedure to be as good or possibly better than the previous procedures.

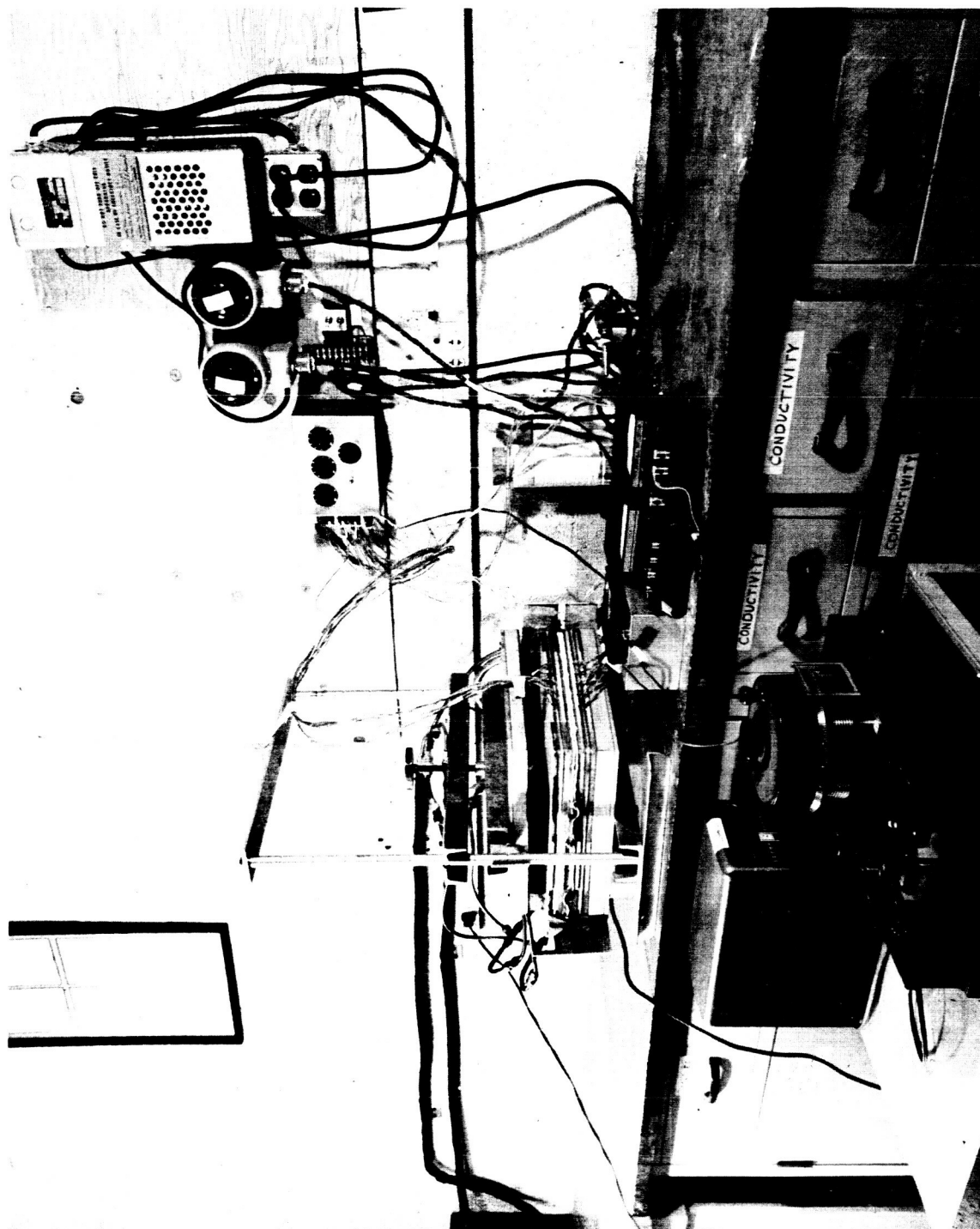


Figure 1. Thermal Conductivity Apparatus

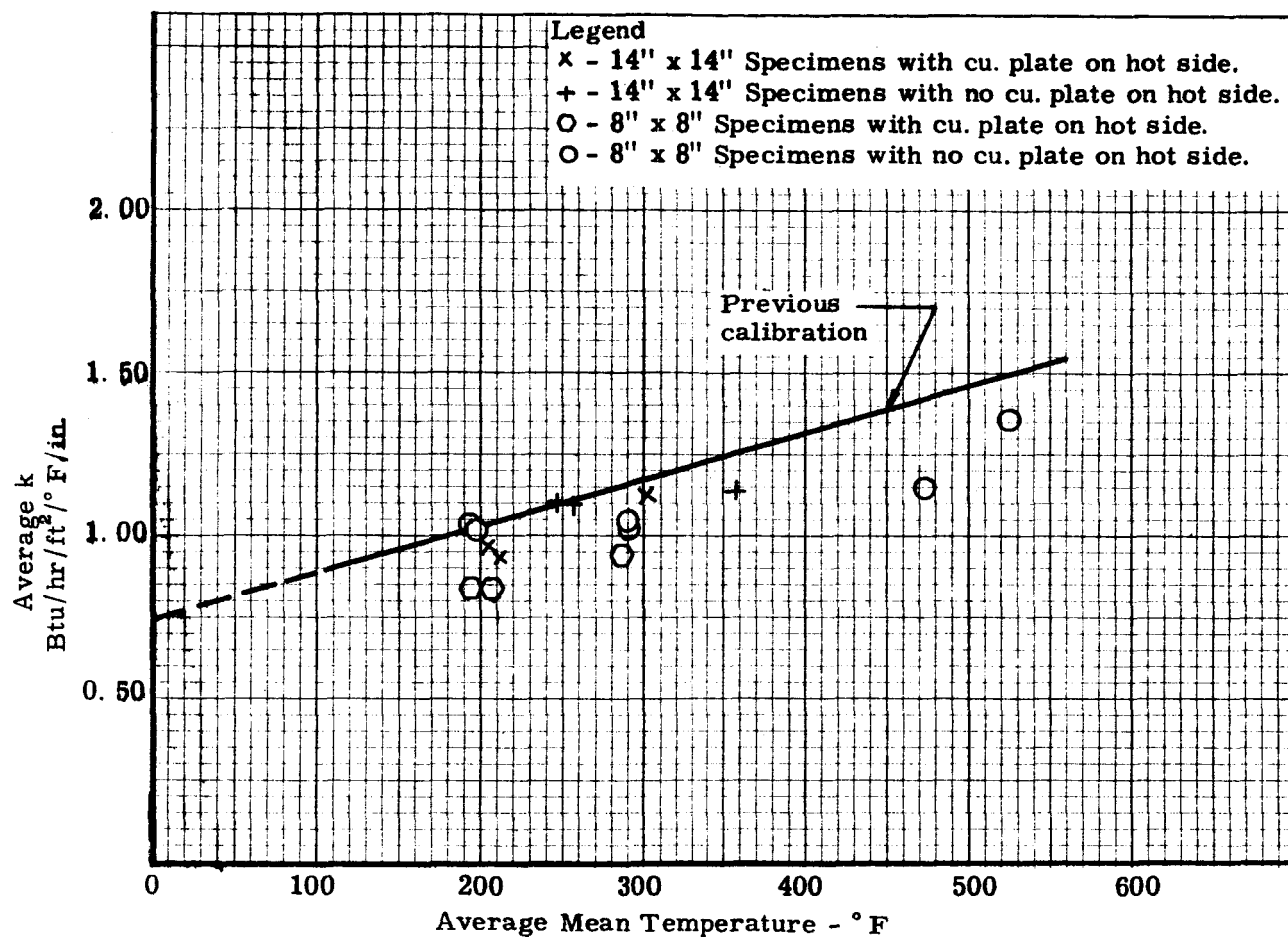


Figure 2. Original Calibration Curve Using Various Size Specimens and Various Measuring Devices.

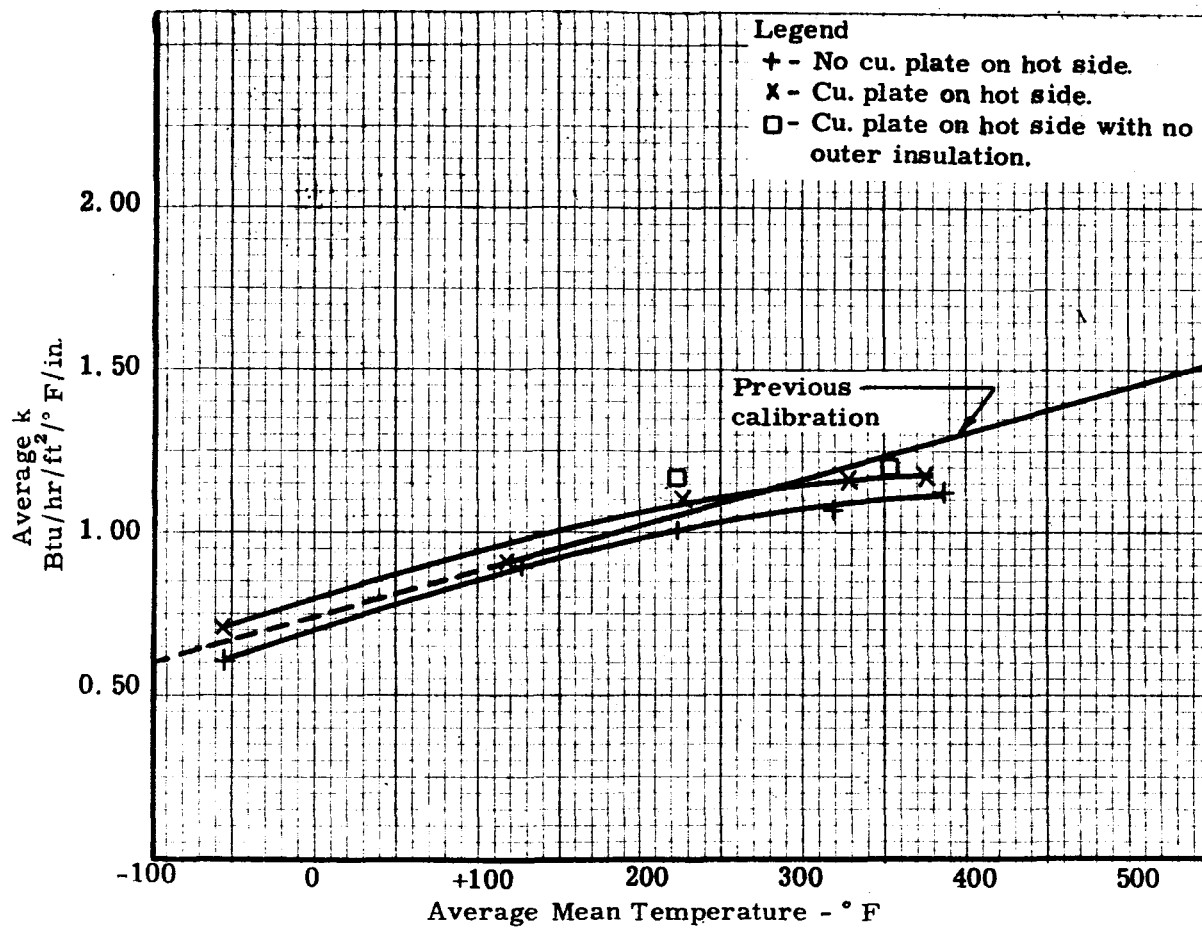


Figure 3. Final Calibration Curve.

✕ SRI Data 14" x 14" x 0.238", asbestos sheets attached to getters on both sides of specimen

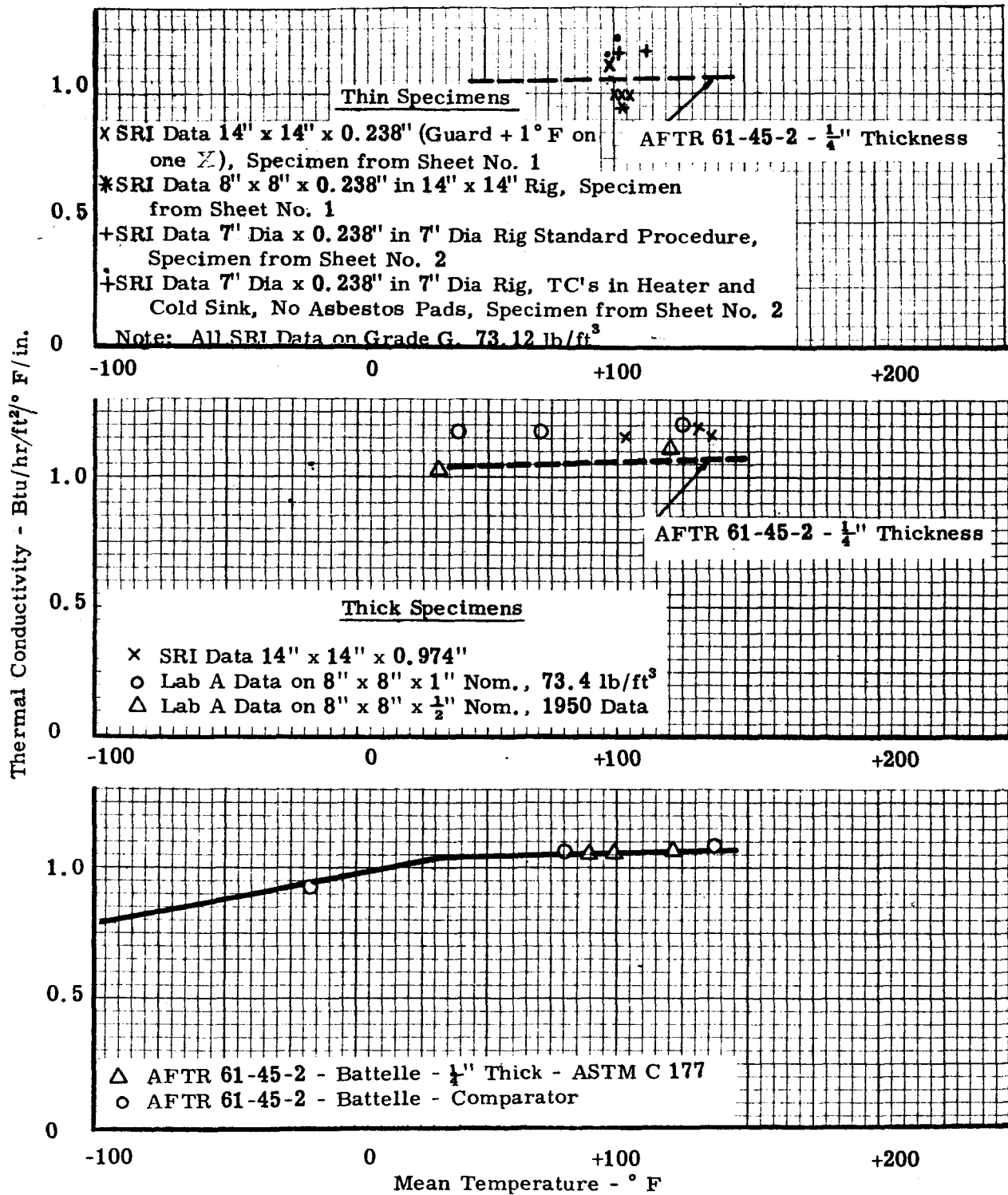


Figure 4. Comparison of Thermal Conductivity of Plexiglas by Different Investigators.

THERMAL CONDUCTIVITY TO 5000°F

The thermal conductivity is determined with a radial heat flow apparatus that utilizes a specimen 1" long. The equipment allows a direct measurement of the thermal conductivity rather than a measurement relative to some standard reference material. A picture of the apparatus ready to be installed in the furnace is shown in Figure 1. The furnace and associated equipment for the thermal conductivity work is shown in Figure 2. In addition to the specimen, the apparatus consists primarily of (1) a water calorimeter that passes axially through the center of the specimen, (2) guards made from the same specimen material at both ends of the specimen to reduce axial heat losses, (3) sight tubes that allow the temperature at selected points in the specimen to be determined either by thermocouples or optical pyrometer, and (4) an external heat source (see Figure 3). The water calorimeter provides a heat sink at the center of the specimen to create a substantial heat flow through the specimen and allows the absolute value of the heat flow to be determined. Thermocouples mounted $\frac{1}{2}$ " apart in the calorimeter water stream measure the temperature rise of the water as it passes through the gage portion of the specimen. By also metering the water flow through the calorimeter, it is possible to calculate the total radial heat flow through the $\frac{1}{2}$ " gage section of the specimen from the standard relationship $Q = WC\Delta T$. W is the weight of water flowing per hour, C is the specific heat of water, and ΔT is the temperature rise of the water as it passes through the gage section.

The standard specimen configuration is 1" long, 1" outside diameter, $\frac{1}{4}$ " inside diameter, with $\frac{5}{64}$ " holes, $\frac{1}{2}$ " deep, on radii of $\frac{7}{32}$ " and $\frac{13}{32}$ ". The $\frac{5}{64}$ " holes in the specimen permit temperature measurement at selected points within the specimen.

A $\frac{1}{2}$ " long upper guard and a 1" long lower guard of specimen material are placed above and below the 1" specimen to maintain a constant radial temperature gradient throughout the entire specimen length and thereby prevent axial heat flow in the specimen. The outer ends of the specimen guards are insulated with graphite tubes filled with thermatomic carbon. These tubes also hold the specimen in alignment. The combined effect of specimen guards and thermatomic carbon insulation permits a minimum axial temperature gradient within the specimen. This gradient is not detectable by optical pyrometer readings. Visual inspection of the specimens after runs have verified that no large axial temperature gradient exists in the specimen. The guards, made of specimen material, display axial distortion of the isothermal lines for approximately $\frac{1}{4}$ " from the outer ends before reaching an apparent constant axial temperature.

The annulus between the specimen inside diameter and the $\frac{7}{32}$ " outside diameter of the calorimeter tube is packed with either copper granules, graphite or zirconia powder. This annulus packing provides a positive method for centering the calorimeter within the specimen and promote good heat transfer between specimen and calorimeter.

On low temperature runs (up to 2000°F), the specimen temperature is measured with Chromel-Alumel thermocouples inserted into the specimen through the sight tubes. At high temperatures, the temperatures are read by optical pyrometer sighting down the sight tube through a right-angle mirror device.

In Figures 1 and 3 showing a typical conductivity calorimeter apparatus ready for insertion into a furnace for a run, a water-cooled stainless steel section can be seen at the top of the unit. This section provides permanent sight tubes to within about $2\frac{1}{2}$ " of the guard specimen, in addition to a permanent mount for the right-angle mirror device used with the optical pyrometer. Within the short zone between the water-cooled section and the top guard, thin-walled graphite sight tubes are fitted. The remainder of the annulus is filled with thermatomic carbon insulation.

During thermal conductivity runs, the following data are recorded: (1) power input, (2) specimen face temperature, (3) specimen temperatures in the gage section at the $\frac{7}{32}$ " and $\frac{13}{32}$ " radii, (4) temperature of the calorimeter water at two points $\frac{1}{2}$ " apart axially within the specimen center, and (5) water flow rate through the calorimeter. At least 3 readings are made at each general temperature range to determine the normal data scatter and to minimize the error that might be encountered in a single reading.

All thermocouple readings are measured on a Leeds and Northrup K-2 null balance potentiometer used in conjunction with a galvanometer of 0.43 microvolts per mm deflection sensitivity. All optically measured temperatures are read with a Leeds and Northrup Type 8622 optical pyrometer. The flow rate of the calorimeter water was measured with a Fischer and Porter Stabl-Vis Flowrator.

The thermal conductivity values are computed from the relation

$$K = \frac{QL}{\Delta T A}$$

where Q is the heat flow to the calorimeter within the specimen gage section, A is the log mean area for the specimen gage length, ΔT is the specimen temperature change across the specimen gage length, and L is the gage length over which the specimen ΔT is measured.

The heat flow Q is determined by the calorimeter. \bar{A} and L are calculated directly for the particular specimen configuration. ΔT is determined directly from the observed temperature difference across the specimen gage length.

Extensive calibration work on materials of known thermal conductivity has indicated that the equipment used in this work generally yields results within 10% of literature values.

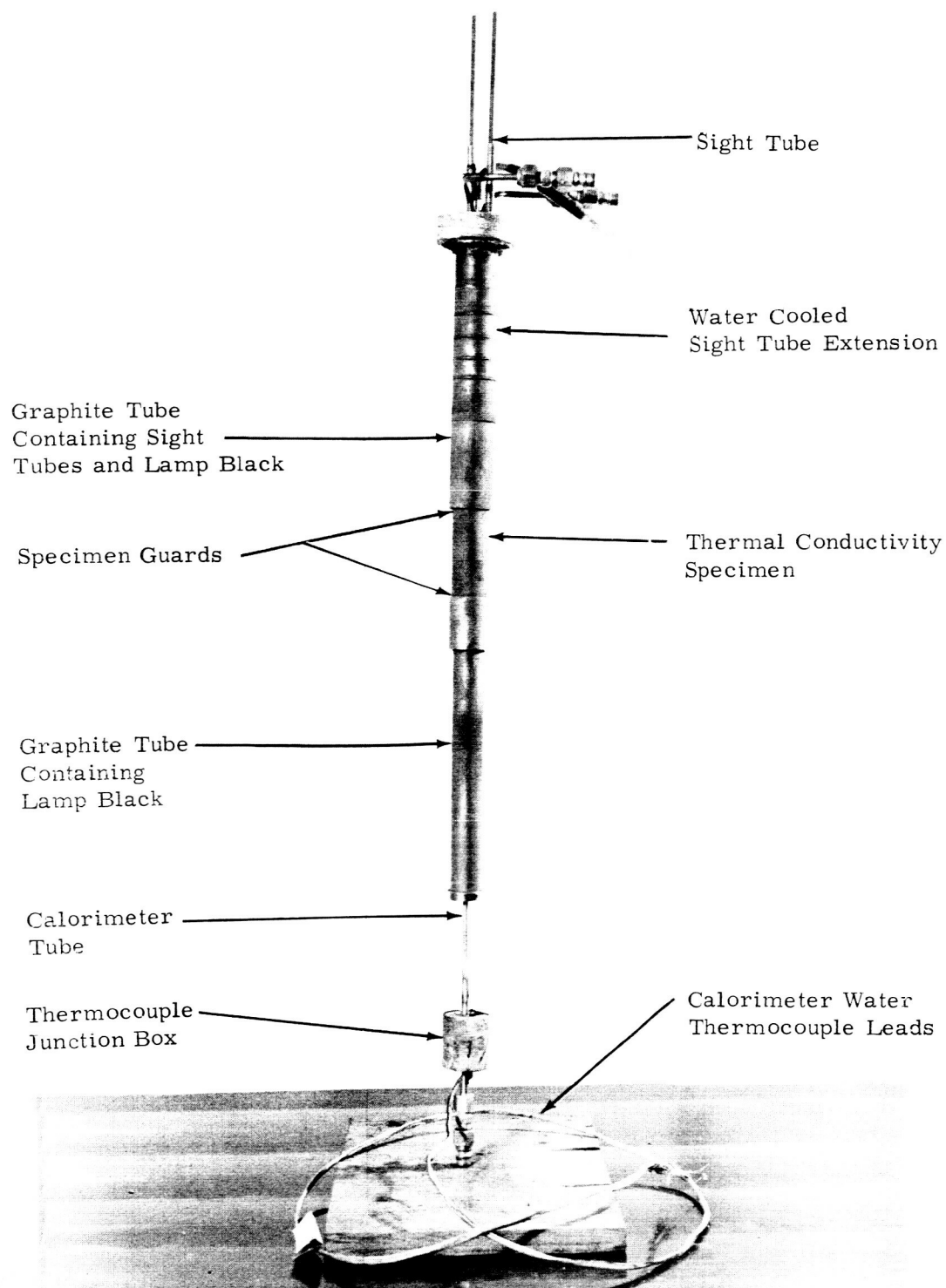


Figure 1. Picture of the Radial Thermal Conductivity Apparatus

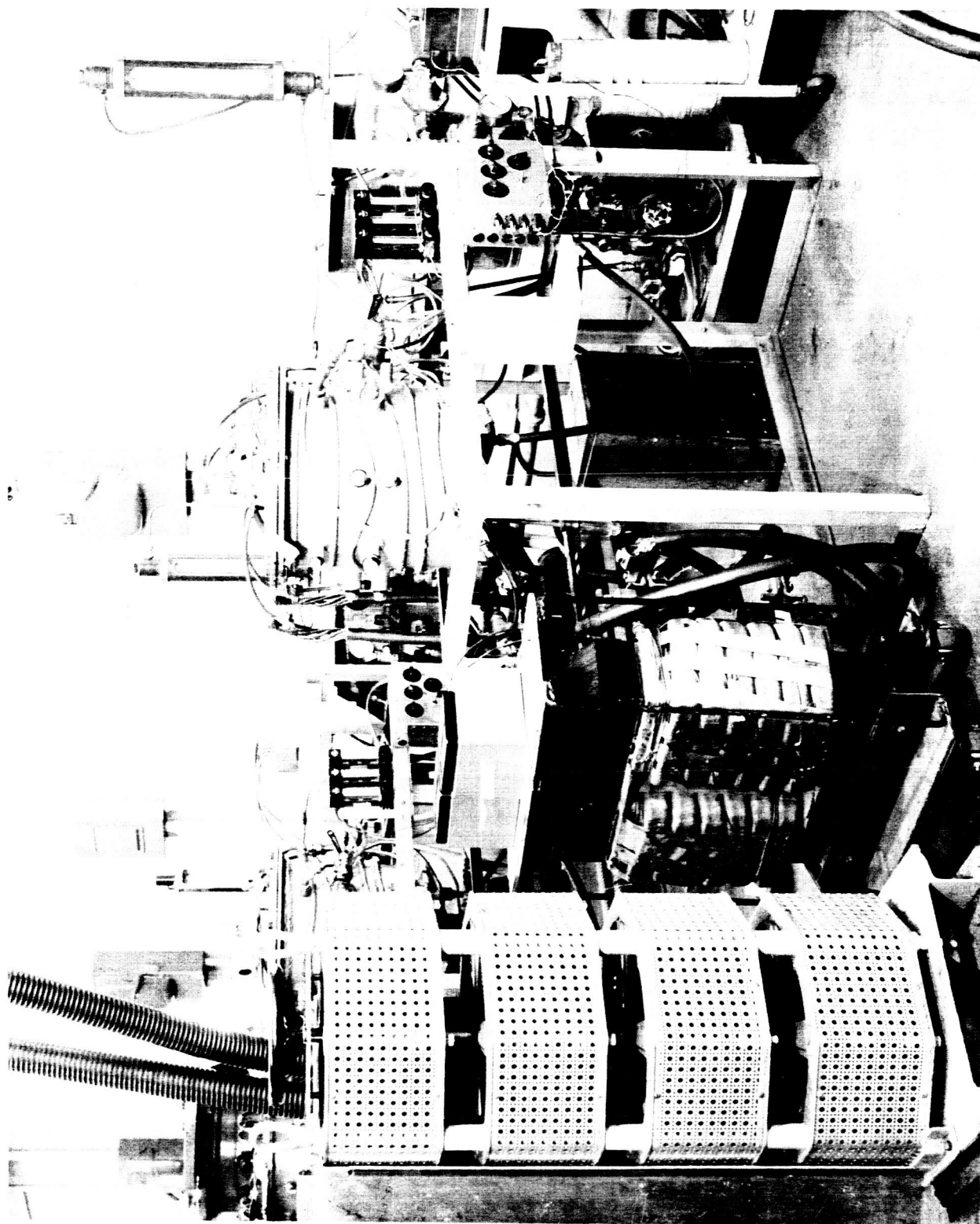


Figure 2. Furnace with Thermal Conductivity Apparatus Installed

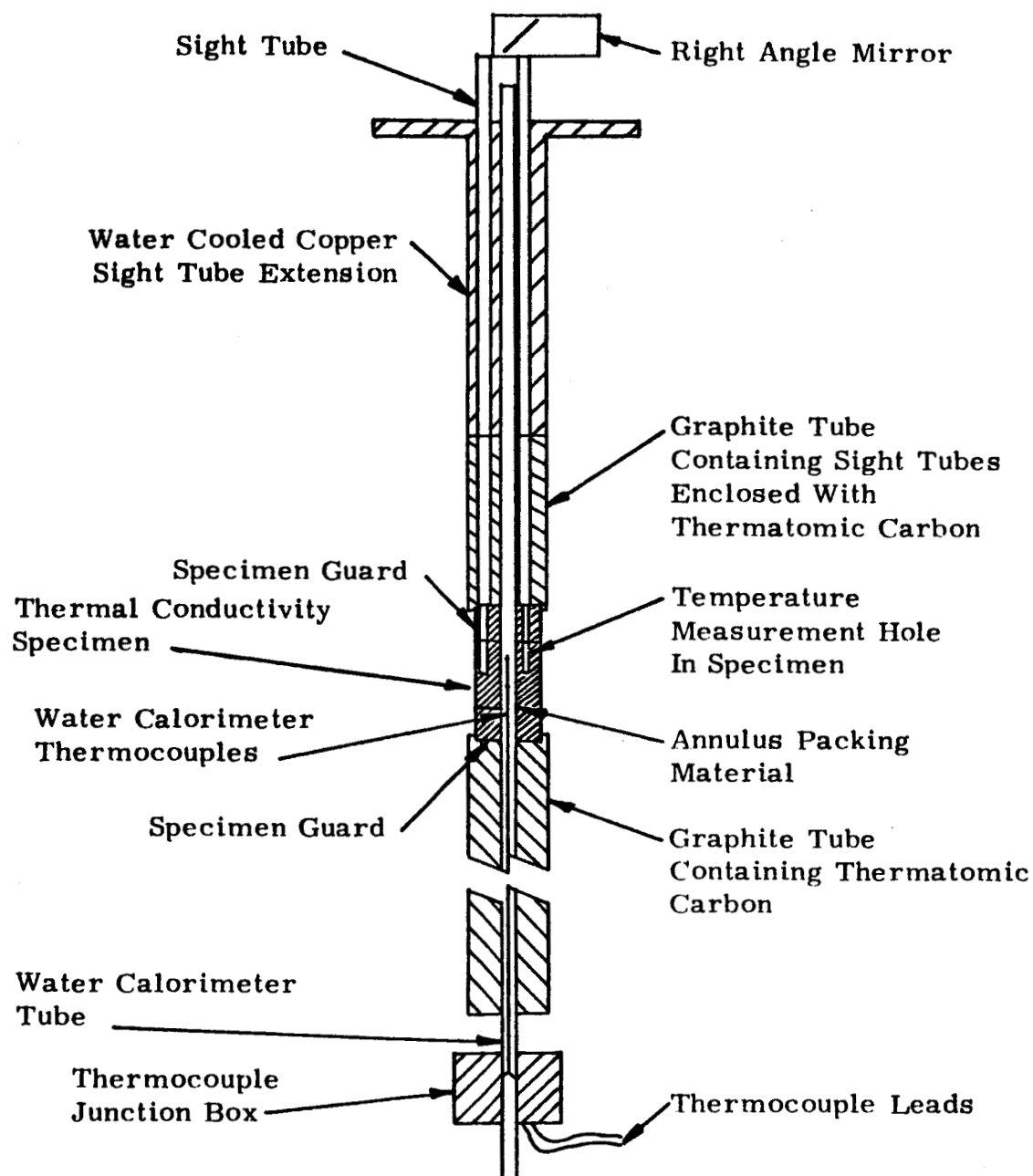


Figure 3. Cross-section Schematic of the Thermal Conductivity Apparatus.